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भारतीय मानक

ग्लाईआक्सल (40 प्रतिशत विलयन) — विशिष्टि

Indian Standard

GLYOXAL (40 PERCENT SOLUTION) — SPECIFICATION

ICS 71.080

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1995

Price Group 3

Organic Chemicals (Miscellaneous) Sectional Committee, PCD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Glyoxal (40 percent solution) and its derivatives are used for the finishing of cotton, viscose, cellulose acetates, wool, silk and polyvinylalcohol fibres because of their high reactivity. Glyoxal is used in making the drug intermediate 2-methyl imidazole (from which metronidazole, tinidazole, cimetidine, etc, are made), in preparing hydantoin, allanatoin, sulphapyrazines, phenylacetamidoglycol aldehyde and certain bactericides.

It is also used in paper coating and insulating paper board, casein binders, curing of stencil duplicating inks and certain cement compositions. It has wide uses in tanning, paint, photography, plastics, tobacco industry, etc.

It has some advantages in use that it does not irritate the skin or mucous membrane. It is quite stable in solution form and can be stored, if protected from heat.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

GLYOXAL (40 PERCENT SOLUTION) — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for glyoxal (40 percent solution).

2 NORMATIVE REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below:

IS No.	*Title*
265 : 1987	Hydrochloric acid (*third revision*)
1070 : 1992	Water for general laboratory use (*third revision*)
2480 (Part 1) : 1983	General purpose glass thermometers : Part 1 Solid stem thermometers (*second revision*)
4905 : 1968	Methods for random sampling

3 REQUIREMENTS

3.1 Description

The material shall be homogeneous, colourless to pale yellow coloured liquid. It is soluble in water, alcohol and acetone.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

4 PRECAUTIONS IN HANDLING

The carboys shall be stored in a cool, well ventilated place.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in polyethylene carboys or as agreed to between the purchaser and the supplier.

5.2 Marking

The material shall be marked with the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net, gross and tare mass of the material; and
d) Lot or batch number, in code or otherwise.

5.2.1 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to the manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn and their conformity to the requirements of this standard be judged as prescribed in Annex B.

Table 1 Requirements for Glyoxal (40 Percent Solution)
(*Clauses* 3.2 *and* B-5.1)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Relative density at 27/27°C	1.26 to 1.30	A-3
ii)	Glyoxal, percent by mass, *Min*	40.0	A-4
iii)	Acidity (as acetic acid), percent by mass, *Max*	0.5	A-5
iv)	*p*H	1 to 3	A-6
v)	Colour, in Hazen Units, *Max*	40	A-7
vi)	Ash content, percent by mass, *Max*	0.1	A-8

ANNEX A

(*Clause* 3.2 and *Table* 1)

METHODS OF TEST FOR GLYOXAL (40 PERCENT SOLUTION)

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 PREPARATION OF SAMPLE

A-2.1 Procedure

Weigh accurately about 2.5 g of the sample and dilute it to 1 litre with water. Use this *prepared sample* for test.

A-3 DETERMINATION OF RELATIVE DENSITY

A-3.0 Outline of the Method

Mass of equal volumes of the material and water are compared.

A-3.1 Apparatus

A-3.1.1 *Relative Density Bottle* — 25-ml capacity.

A-3.1.2 *Water Bath* — Maintained at 27±0.2°C.

A-3.1.3 *Thermometer* — Any convenient thermometer of a suitable range with 0.1 degree or 0.2 degree graduation [*see* IS 2480 (Part 1) : 1983].

A-3.2 Procedure

Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water at 27°C. Fill to cover flowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in the water bath, keep the entire bulb completely covered with water and maintain the bath temperature at 27 ± 0.2°C for 30 minutes. Remove the relative density bottle from the bath. Wipe completely, dry and weigh. Calculate the mass of water. Again cool and carefully dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-3.3 Calculation

$$\text{Relative density at } 27°\text{C}/27°\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle with the material;

B = mass in g of the relative density bottle; and

C = mass in g of the relative density bottle with water.

A-4 DETERMINATION OF GLYOXAL CONTENT

A-4.0 Outline of the Method

Glyoxal is quantitatively oxidized to formic acid by sodium periodate solution. The formic acid formed is determined by titrating it against standard sodium hydroxide solution using methyl red indicator. Glyoxal content is corrected for free acids present by running a blank titration without sodium periodate solution.

A-4.1 Reagents

A-4.1.1 *Sodium Periodate Solution*

Dissolve 21 g of sodium periodate in water. Add 5 ml of methyl red indicator and neutralize the solution to just yellow by titrating it against 0.1 N NaOH solution. Dilute this neutralized solution to 1 litre with distilled water.

A-4 1.2 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-4.1.3 *Methyl Red Indicator Solution* — 0.1 g in 100 ml of 60 percent alcohol.

A-4.2 Procedure

Pipette out 50 ml of the freshly prepared sodium periodate solution in a 250-ml flask. Add 10 ml of *prepared sample* (*see* **A-2**) by pipette. Keep the reactants at room temperature for 45 minutes. Titrate against standard 0.1 N NaOH solution to yellow end point (V_1 ml).

Pipette out 10 ml of the *prepared sample* (*see* **A-2**) in another 250-ml flask. Add about 40 ml of distilled water. Titrate this solution against standard 0.1 N NaOH using methyl red as an indicator to yellow end point (V_2 ml).

A-4.3 Calculation

Glyoxal content, percent by mass

$$= \frac{N \times (V_1 - V_2) \times 2.902}{0.01 \times M}$$

where

M = mass in g of the material taken for the test (*see* A-2);

N = normality of standard sodium hydroxide solution;

V_1 = volume in ml of standard sodium hydroxide for sodium periodate titration; and

V_2 = volume in ml of standard sodium hydroxide solution for blank titration.

A-5 DETERMINATION OF FREE ACIDITY

A-5.1 Reagents

A-5.1.1 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-5.1.2 *Phenolphthalein Indicator*

Dissolve 1 g in 60 ml of alcohol and dilute with water to 100 ml.

A-5.2 Procedure

Pipette out 25 ml of the *prepared sample* (*see* **A-2**) in a 250-ml conical flask. Add few drops of phenolphthalein indicator. Dilute with water to approximately 100 ml and titrate against 0.1 N NaOH solution to a pink end point.

A-5.3 Calculation

Acidity (as acetic acid), Percent by mass

$$= \frac{V \times N \times 6.003}{0.025 \times M}$$

Where

V = volume in ml of standard sodium hydroxide solution,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test (*see* **A-2**).

A-6 DETERMINATION OF *p*H

A-6.1 Procedure

Determine *p*H of the material at 27 ± 0.2°C with the help of a suitable *p*H meter, using glass electrode.

A-7 DETERMINATION OF COLOUR

A-7.0 Outline of the Method

The colour of the samples is compared with that of permanent colour standard and expressed in terms of Hazen colour units.

NOTE — The Hazen colour unit is defined as the colour of an aqueous solution containing one part per million of platinum in the form of chloroplatinic acid and in the presence of 2 parts per million of cobaltous chloride ($CoCl_2.6H_2O$)

A-7.1 Apparatus

A-7.1.1 *Flat-Based Colorimetric Tubes*

Approximately 20 mm external diameter and having a graduation mark 100 mm above the base.

A-7.2 Reagents

A-7.2.1 *Cobaltous Chloride, Hexahydrate*

A-7.2.2 *Hydrochloric Acid* — conforming to IS 265 : 1987

A-7.2.3 *Chloroplatinic Acid*

Dissolve 250 mg of platinum in a small quantity of *aqua regia* contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

A-7.2.4 *Potassium Chloroplatinate* K_2PtCl_6 — 1.25 g of the salt containing 0.5 g of platinum.

A-7.3 Preparation of Colour Standards

A-7.3.1 Dissolve 0.50 g of cobaltous chloride, hexahydrate and whole of chloroplatinic acid (**A-7.2.3**) in 50 ml of hydrochloric acid. Warm, if necessary, to obtain a clear solution, and after cooling, pour into the 500-ml volumetric flask. Dilute with water up to the graduation mark.

A-7.3.2 From this solution prepare a series of permanent colour standards ranging from zero Hazen colour unit upwards, at interval of 10 units. For each 10 units, pipette 5.0 ml of the solution into the 250-ml volumetric flask and dilute with water up to the graduation mark. Transfer each standard solution to a storage bottle. Check these standards at intervals against freshly prepared standards.

A-7.4 Procedure

Pour into the colorimetric tube a quantity of the sample sufficient to fill it to the graduation mark. Similarly pour the colour standard selected into the other tube to the mark. Compare the colour of the sample with that of the standard by looking down the tubes against a white background, taking care to avoid side illumination. Repeat with other colour standards, if necessary, until the closest match is obtained.

A-7.5 Report

Report the result to the nearest 10 Hazen colour units.

A-8 DETERMINATION OF ASH CONTENT

A-8.1 Procedure

Evaporate about 10 g of the material, accurately weighed, to maximum possible level in a weighed platinum or silica basin, and gently ignite the residue until all carbonaceous matter has disappeared. Cool in a desiccator and weigh.

A-8.2 Calculation

$$\text{Ash, Percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass of the ignited residue, and

M_2 = mass of the material taken for the test.

ANNEX B

(*Clause* 6.1)

SAMPLING FOR GLYOXAL (40 PERCENT SOLUTION)

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken at a place protected from damp, air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible, by suitable means.

B-1.5 The samples shall be placed in a clean, dry and airtight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, year of manufacture and other important particulars of the consignment.

B-1.8 Samples shall be stored in a cool and dry place.

B-2 SAMPLING INSTRUMENT

B-2.0 The following forms of sampling instrument may be used:

a) Sampling bottle or can for taking samples from various depths in large tanks, and
b) Sampling tube.

B-2.1 Sampling Bottle or Can

It consists of a weighted bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 1). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

B-2.2 Sampling Tube

It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

B-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3 SCALE OF SAMPLING

B-3.1 Lot

In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group constitute a separate lot.

B-3.2 Tests shall be conducted on each lot, separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

B-3.3 The containers shall be selected at random from the lot. In order to ensure the randomness of selection, the method given in IS 4905 : 1968 may be followed.

Table 2 Scale of Sampling

(*Clause* B-3.2)

Lot Size N (1)	No. of Containers to be Selected n (2)
Up to 100	5
101 to 200	6
201 to 300	7
301 to 400	8
401 to 500	9
501 and above	10

NOTE — In the case of very small lots where the selection of five containers may be uneconomical, the number of containers to be selected and the method of judging the conformity of the lot to the requirements of this specification shall be as agreed to between the purchaser and the supplier.

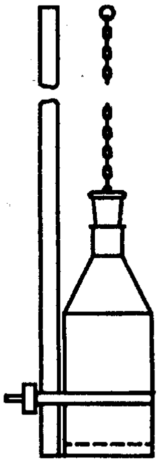
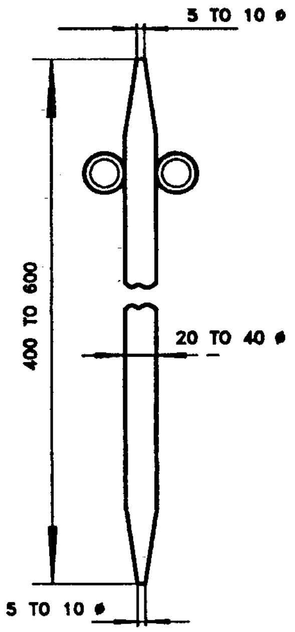


FIG. 1 SAMPLING BOTTLE OR CAN



All dimensions in millimetres.

FIG. 2 SAMPLING TUBE

B-4 PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected as in **B-3.2** a representative portion of the material from different parts of the container shall be drawn with the help of a suitable instrument (*see* **B-2**).

B-4.2 From each of these individual portions (*see* **B-4.1**) an equal quantity of material shall be taken and thoroughly mixed to constitute a composite sample not less than 1 000 ml in volume. The composite sample shall be divided into three equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample. These shall be transferred to separate containers and shall be sealed and marked with full indentification particulars given under **B-1.7**.

B-5 TESTS

Tests for the determination of all the characteristics given in **3.1** and Table 1 shall be conducted on the composite sample.

B-6 CRITERIA FOR CONFORMITY

For declaring the conformity of a lot to the requirements of all the characteristics, the test result for each of the characteristics shall satisfy the corresponding requirement specified in the standard.

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This Indian Standard has been developed from Doc : No. PCD 9 (966).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Dee Kay Printers, New Delhi-110015, India